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TECHNICAL NOTE

D-906

INVESTIGATIONS OF THE DETERIORATION OF
22 REFRACTORY MATERIALS IN A MACH NUMBER 2 JET AT A
STAGNATION TEMPERATURE OF 3,800° F

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SUMMARY

A limited investigation of the deterioration characteristics of 22 refractory materials was conducted by exposing them to a stagnation temperature of 3,800° F in a Mach number 2 ceramic-heated jet at the Langley Research Center. The materials tested were six materials whose major constituent was silicon carbide, five cermets whose major constituent was titanium carbide, six materials whose major constituents were metal borides, four cermets containing alumina, and one silicon nitride model. Tests consisted of obtaining weight change and appearance changes for 1/2-inch-diameter hemispherical-nose cylindrical models exposed to the air jet for 30 seconds at a time for a total of four runs or 2 minutes exposure. Curves of weight changes plotted against the number of 30-second tests in the jet were obtained. Estimates of average surface temperature near the stagnation point of the model were obtained by use of a special temperature-measuring camera. The models were examined before and after the completion of the tests for possible changes in microstructure; no significant changes were found. The data obtained were analyzed with the view that the oxidation characteristics of the materials were the main factor in deterioration of the materials under the conditions of the tests. It was concluded that only those materials which changed in weight the least could be recommended for further extensive application-oriented evaluations. The following materials fell in this category: silicon carbide - silicon, chromium - 28-percent alumina cermet, titanium boride - 5-percent boron carbide. The remainder of the materials tested had oxidation characteristics which appeared to be too severely limiting of their general applications to flight vehicles.

INTRODUCTION

The structures and skins of hypersonic vehicles may be aerodynamically heated in flight to temperatures above 2,000° F. The materials which may be used for these vehicles must be able to withstand the high

temperatures without significant loss of physical and mechanical properties. The deterioration of refractory materials other than oxides in air at high temperatures is considerably influenced by the oxidation characteristics of the materials. In many cases it is the poor high-temperature oxidation resistance which limits the usefulness of otherwise excellent materials. It is not sufficient to test the materials at high temperatures in quiescent air because many materials form oxides which may be somewhat protective under these conditions but not under the flight conditions of high speed where mechanical erosion may continuously remove the oxides. Whereas most of the refractory materials are themselves fairly resistant to mechanical erosion, the oxides are generally much less resistant. Thus the oxidation characteristics of the material is considered to be the main factor involved in the deterioration of the refractory materials of this investigation. A material which loses weight rapidly is obviously becoming less useful due to its decrease in thickness and resultant depletion of mechanical properties. A material which gains weight rapidly on oxidation can be just as seriously changed in mechanical properties since the oxide coating has generally poor mechanical properties and the original material is decreasing rapidly in thickness also. In cases where dimensional stability or surface smoothness is necessary neither of these types of materials will prove satisfactory. Further, in many applications it would not be desirable to overdesign by allowing for loss of weight in one case or loss of properties for oxidation where gain in weight occurs. From this it follows that the most useful materials will be the ones which change in weight and surface condition the least.

In order to obtain some estimates of the extent of deterioration under conditions of high temperature and supersonic or hypersonic flow, a number of refractory materials having excellent thermal shock resistance properties were selected for testing in the Mach 2, 3,800° F stagnation temperature ceramic-heated jet (ref. 1) at the Langley Research Center. Materials were selected as those whose general physical properties made them candidates for nose and leading-edge materials for hypersonic vehicles. These materials cover a range of densities, specific heats, and thermal conductivities which will affect their utility in any given application. Table I lists these materials, their sources, and other information of interest. Hemispherical-nose cylinder models 1/2 inch in diameter were obtained for the tests. The scope and experimental conditions of the tests were governed by the capabilities of the ceramic-heated air jet and the limited number of models available. The tests in the jet yielded weight-change data, approximate surface temperatures near the model stagnation point and a qualitative evaluation of appearance changes. Internal-microstructure-change examinations were made by metallographic techniques. These results offer information of a screening nature to permit the selection of a few materials for further qualification tests for specific application to the design of structures for hypersonic vehicles.

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MATERIALS AND MODELS

The materials were selected from a large number which had been evaluated in reference 2 for application as flameholders. Only materials which had the best thermal-shock resistance were selected. In all, 22 materials were selected: 6 had silicon carbide as the major constituent, 5 were cermets whose major constituent was titanium carbide, 6 were metal borides, 4 were alumina cermets, and there was 1 silicon nitride model. Some of the materials were quite similar to each other but had been made by different processes or agencies. Table I lists the materials along with source and designations. Approximate compositions and other data are given where available. Models were machined in the shape of hemispherical-nose cylinders of 1/2-inch diameter. Figure 1 shows a 1/2-inch-diameter model during test. The models were held in a conical graphite adapter on the end of a steel sting during the jet runs.

TEST FACILITY AND OTHER APPARATUS

Laboratory-Scale Ceramic-Heated Air Jet

This facility produces a test jet of heated air with a velocity of Mach number 2 and an approximate maximum stagnation temperature capability of 4,000° F. The stagnation pressure is 105 lb/sq in. The useful portion of the jet is within a jet column of circular cross section of about 5/8-inch diameter. The air is heated by a reverse flow through a ceramic-pebble bed which is first heated to the desired temperature. Details of the construction and use of this facility are contained in reference 1.

Laboratory Apparatus

The models were weighed on an analytical balance to the closest milligram.

Surface temperature estimates of the models while in the jet stream were made using a modified K-24 aerial camera and film densities were read with a densitometer. Details of the camera and the method of estimating temperature by its use are contained in reference 3.

Microstructure examinations were made by use of Buehler metallographic specimen preparation equipment and Bausch and Lomb research metallographic equipment.

EXPERIMENTAL PROCEDURES

Jet Test Procedure

The models were weighed to the nearest milligram and mounted in the remotely controlled mechanism for inserting the model in the jet. The pebble bed of the heater was heated to about $4,000^{\circ}$ F. The heating equipment was shut off and the pressurized air was forced in reverse flow through the bed and through the Mach number 2 nozzle. The model was inserted directly over the nozzle opening and received the direct blast of the hot air jet. The model was exposed to the jet for 30 seconds and then removed. The air was shut off. The pebble bed temperature (usually about $3,600^{\circ}$ F) was read by optical pyrometer as soon as possible (approximately 1 minute). Stagnation temperature was taken as the average between the initial and final bed temperature readings. After cooling under ambient conditions, the model was removed from the sting at room temperature and was weighed again. This last weight was taken as the initial weight for the next run of that model unless the next run for that particular model was delayed for a few weeks. When there was a considerable time lag between runs, the models were reweighed just before the next run. Only in a very few cases was a change in weight observed under these conditions and then the data were corrected. Observations were made on the appearance of the model nose as to color, nature of oxide or glaze, and evidence of erosion.

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Color motion-picture and temperature cameras were operating during the run. The temperature camera films were read by measuring the density of the blackened image of the model at a location as close to the stagnation point as possible. These densities were compared to calibration charts relating density, exposure, and temperature in order to obtain the surface temperatures. Temperature data of the model surface were taken on an average of about every 2 seconds during the runs. Figure 2 shows typical curves of 2-run average camera temperatures near the model stagnation point plotted against time in the jet. The motion pictures of the runs were taken, in most cases, without any artificial lighting because the temperature camera required a relatively dark background in order to make the measurements of the model. Thus, the motion pictures showed only the glowing nose of the model.

Microscopic Examination Procedure

For the microscopic examination of the models, a slice of the material was cut off the back end before jet tests. After the jet tests the model was cut longitudinally on the center line, and was cut perpendicular to the length at approximately the base of the hemisphere of the nose. The specimens were then mounted to expose internal surfaces

in both the longitudinal and lateral directions of the tested model and the lateral direction of the untested specimen. The specimens were then polished and etched and photomicrographs were made of these surfaces. The specimens also were examined visually. These microscopic examinations were performed to determine whether any observable changes occurred to the internal microstructure such as a change in grain or crystal size or whether changes occurred in the material close to the model nose surface as a result of the jet tests.

Quantitative Significance of the Data

The jet conditions were not known precisely. The jet-pebble-bed temperature measurements were taken about 30 to 60 seconds before the model went into the jet and 60 to 90 seconds after the model came out of the jet. The average of these two optical pyrometer measurements of temperature of top surface of the ceramic pebble bed was called the average stagnation temperature. Thus, the actual jet temperature was not measured or accurately known. Unpublished temperature-calibration data of this facility indicate that the average temperature as obtained from the optical pyrometer measurements mentioned previously is a maximum and that the actual stagnation temperature may be as much as 200° F lower. For the comparisons of weight-change behavior in this investigation it was assumed that all runs were made under essentially the same jet conditions. The model weights were taken on an analytical balance the precision of which is about ± 0.2 milligram. There was some ceramic dust in the jet from time to time which adhered to the model nose. Most of the time this material could be removed easily and was removed before weighing. An investigation of the quantity of dust adhering to the model noses showed that it varied from run to run and that as much as 10 milligrams could accumulate. However, since most of this dust was removed before weighing a model, it is believed that changes in weight greater than 2 milligrams are significant and due to other causes than adhering ceramic dust. It is believed that the weight changes were chiefly due to oxidation effects including loss of oxides by mechanical erosion as well as formation of volatile oxides or solid oxide scales.

RESULTS AND DISCUSSION

The data of the weight-change studies of the models in the laboratory-scale ceramic-heated jet at an average stagnation temperature of 3,800° F and the final condition of the model noses are given in table I. Also included under test results in table I are the average surface temperatures near the stagnation point of the model during the tests as obtained by a camera. The numerical average of all points and runs for a given

model is the value given in table I. Figure 2 shows typical, average camera temperature curves as a function of time for models M-7 and M-19 which indicate that the surface temperature was essentially constant over about the last 25 seconds of the 30-second runs. The weight change of the model as a variation with number of 30-second tests in the jet appears in figures 3 to 6 where materials having a common major component are plotted on the same figure.

In considering the weight-change data, it appears that in many cases the change for the first 30-second jet test is quite different than that for the last three 30-second tests. This seems to indicate that the first run changed the original condition of the model and that subsequent runs were tests of the model in the altered condition. It would appear logical then in making comparisons to consider only the weight-change trends for the last three runs of each model. The changes which take place during the first run should not be ignored, however, as they may represent changes which will occur in use unless the material is preconditioned.

The results obtained for most of these materials appear to be approximately explained by consideration of known oxidation characteristics of the components present. Classifying the materials according to major component seems to be the best procedure for discussing this facet of the work. Those materials containing silicon carbide (SiC), M-1 to M-6 (see, fig. 3 and table I), would normally be expected to react slightly with oxygen to form a volatile oxide of carbon (CO or CO_2) and the glassy oxide of silicon (SiO_2) which should result in a net gain in weight. However, at these temperatures the glass is liquid enough to be blown away in the jet stream. Thus, a net loss is to be expected and this was observed for all of those models except M-5 (silicon carbide - silicon) which showed no net change. When the other component is considered, further weight changes may be predicted. For the models containing carbon a net loss is expected due to the volatile oxides formed. Materials M-2, M-3, and M-4 contained carbon as well as SiC and all of these lost more than M-1 which was only SiC . Those models which contained SiC and a component other than carbon, that is, M-5 with Si and M-6 with silicon nitride (Si_3N_4) should gain weight if the glassy SiO_2 remains. M-5 showed essentially no weight change yet had slight surface erosion. This can best be explained as a balance in weight gain due to oxidation and loss due to mechanical erosion, both being slight. In the case of M-6, the model lost a significant amount of weight in the first 30-second run and in subsequent runs gained slowly in a fairly uniform manner. The behavior of M-6 was quite similar to that of M-7, which was pure Si_3N_4 , except that the initial loss was much less for M-7. The reason for the initial loss in these two cases is not known.

The next class was composed of cermets containing titanium carbide (TiC) and a metal or alloy and included materials M-8 to M-12. (See, fig. 4 and table I.) TiC was the major component and its oxidation would yield volatile oxides of carbon and solid yellowish titanium dioxide (TiO_2) which should give a net increase in weight if the TiO_2 remains on the surface. M-8 containing 10 percent nickel (Ni) and M-9 containing 20 percent Ni initially showed an increase in weight and then started to lose, both tendencies being slight. The loss may have been due to loss of the TiO_2 by erosion due to the flowing air. However, the solid coating was not yellow but brown which indicates that the Ni may have been oxidizing too which, with its blackish oxide, NiO , would give the brown color observed. These two oxides probably were being formed at the same time and were not adherent enough to remain. The remaining models containing TiC were M-10 containing 40 percent Ni, M-11 containing 40 percent nickel-molybdenum alloy (Ni-Mo), and M-12 containing 50 percent Ni-Mo alloy. All of these last three lost weight quite rapidly and there was evidence of a melting of the oxide products. It is known that Mo often causes an increase in oxidation rate of alloys and thus it could have been expected that M-11 and M-12 would deteriorate fairly rapidly. There seemed to be a trend also for greater rates of deterioration of these types of cermets with increasing amounts of Ni indicating that TiC by itself might be more oxidation resistant than any of these cermets.

The next major group was those which contained metal borides. (See fig. 5 and table I.) Materials M-13, M-14, M-15, and M-18 were pure borides, the first three of which were titanium boride (TiB_2) and the last chromium boride (CrB_2); M-16 was a titanium boride (TiB_2) - 5 percent boron carbide (B_4C) model, and M-17 was a TiB_2 - Ni model. One would normally expect all of these to gain weight since only solid products of oxidation are expected in the temperature range covered with exception of B_4C , a minor constituent of M-16. It is expected that glassy boron oxides will be formed and depending on the amount may become liquid and flow back and possibly be blown away or boil away. Several of the models had acquired a slight glassy coating and in a few instances there was evidence of flow back from the nose of the model. All of these models gained weight in the jet runs except M-15 which initially lost weight in the first 30-second run and then gained at approximately the same rate as the other TiB_2 models in the last three runs. This apparently anomalous behavior of M-15 has not been explained. With the metal borides, the addition of small amounts of other material seemed to give resistance to oxidative deterioration, for example, M-16, TiB_2 - 5 percent B_4C , had the least weight change and M-17, TiB_2 -Ni, was next best, both being significantly more resistant to deterioration due to oxidation than the pure TiB_2 models or the CrB_2 which was the least resistant as shown in figure 5.

The remaining group of materials was cermets containing alumina (Al_2O_3) and a metal or alloy phase. (See fig. 6 and table I.) M-19 and M-20 were Cr- Al_2O_3 cermets, M-21 was a Cr-Mo alloy- Al_2O_3 cermet, and M-22 was a chromium-tungsten alloy-alumina cermet (Cr-W alloy- Al_2O_3 cermet). For these types of materials the oxidation characteristics should be similar to that for the metal or alloy only since Al_2O_3 is already in its highest oxidation state. Thus it would be expected that M-19 and M-20 would be the most resistant to oxidation and would gain slightly forming a fairly adherent oxide coating. This is what occurred and for some unknown reason M-19 gained significantly more than M-20. For the models containing the Cr-Mo or Cr-W alloy it would be expected that there should be less oxidation resistance and probably an overall loss in weight due to the effect of Mo or W which generally catalyze oxidation of alloys and form volatile oxides. Model M-21, however, gained weight in these tests which was contrary to the expected. M-22, the Cr-W alloy - Al_2O_3 cermet, performed as expected by losing considerably in weight.

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In general, the composite-type refractories which usually have physical properties that are somewhere in between those of the single components, have chemical properties approximately the same as the individual components and which probably are quantitatively controlled by the concentrations at the reacting surface. As has been noted, many of the materials tested exhibited a change in weight for the first 30-second run which was inconsistent with the changes that occurred for the subsequent three runs. The reason for this is thought to be that the model surface was altered in chemical composition by the initial oxidation process to the extent that a different oxidation mechanism was responsible for the weight changes of the last three runs compared to the first run.

The fact that all of the models, at room temperature, were inserted into the jet and heated to equilibrium temperature in about 5 seconds and then allowed to cool under ambient conditions to room temperature again for 4 cycles without apparent damage, confirmed their good general resistance to thermal-shock damage.

No figures or data are given of the microscopic studies of the internal microstructure because the examinations revealed essentially no changes in microstructure had occurred due to the testing.

CONCLUSIONS

The results of these studies concern the weight change of the various materials and the relationship of these changes to possible usefulness of the materials for hypersonic missile and reentry vehicle

construction. The nature of the results is mainly directed toward elimination of some of the candidate materials and the recommendation of others for further investigations in application-oriented tests.

The following conclusions have been reached:

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1. Materials which showed the best resistance toward deterioration in these tests were: silicon carbide - silicon, chromium - 28 percent alumina cermet, titanium boride - 5 percent boron carbide. It is believed that these materials should be tested for specific applications where better materials are required for high-temperature, supersonic airstream exposure, as their useful life expectancy is greatest. The silicon carbide - silicon material has an advantage over the other two in its lower density.

2. The following materials are not considered suitable for use in situations where high aerodynamic heating occurs: silicon carbide - carbon composites, chromium-tungsten - alumina cermets, chromium boride, titanium carbide - nickel cermets of nickel content greater than 20 percent, titanium carbide - nickel-molybdenum cermets. These, in general, change in weight much too rapidly to have sufficiently useful life expectancies.

3. The following materials may be useful for specific applications where their type of deterioration is not particularly serious or long life is not of primary importance: chromium-molybdenum - alumina cermet, titanium carbide - nickel cermet when nickel content is 20 percent or less, silicon carbide, titanium boride, titanium boride - nickel, silicon carbide - silicon nitride, and silicon nitride. These would have to be further qualified in specific tests.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Field, Va., April 7, 1961.

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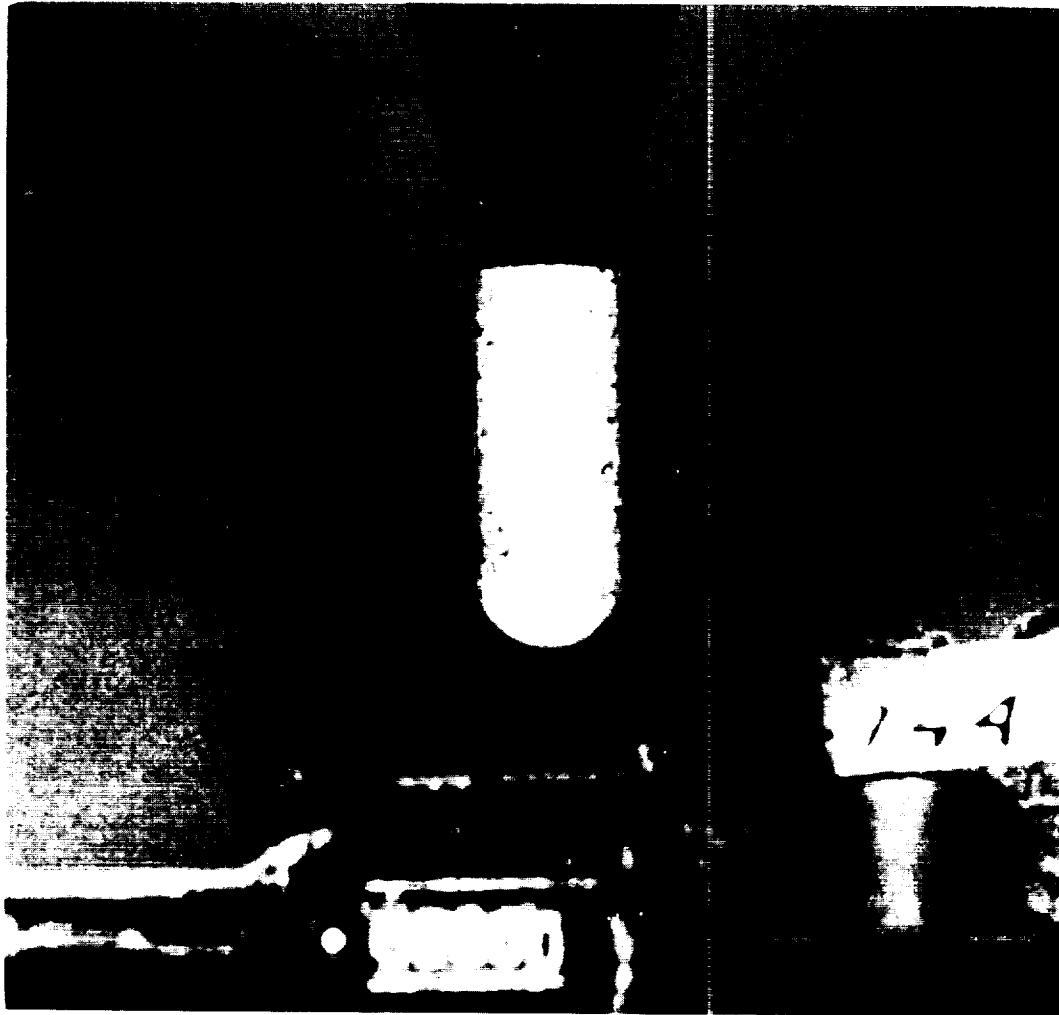
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TABLE 1.- DESCRIPTION OF MATERIALS AND RESULTS OF THE TESTS OF 1/8-INCH-DIAMETER MODELS IN A MACH NUMBER 2 JET AT A STAGNATION TEMPERATURE OF 3,800° F

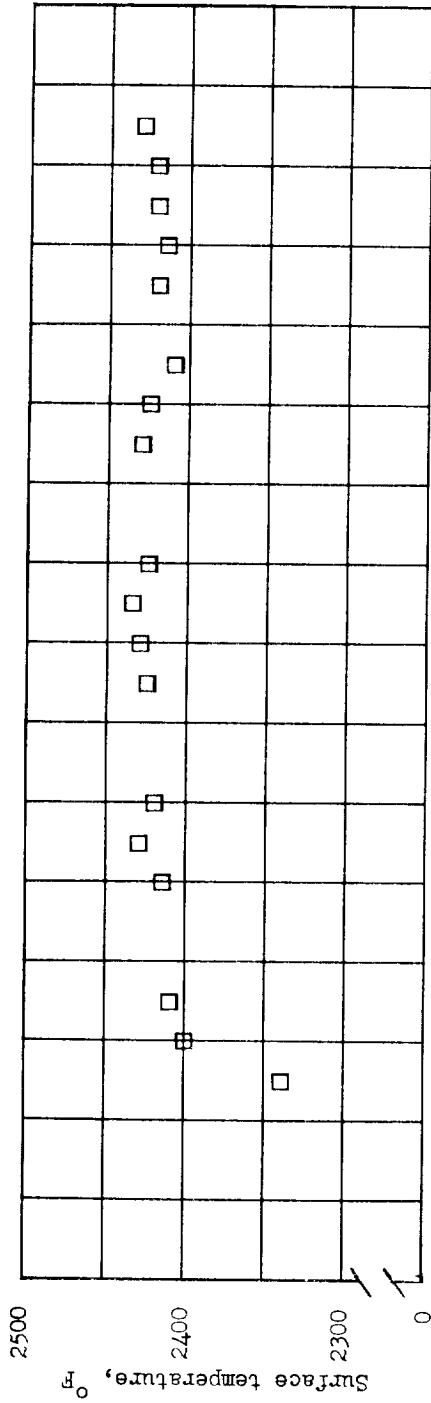
Model	Material and designation	Source	Properties				Test results						
			Density, lb/cu ft	Specific heat, Btu/lb-°F	Thermal conductivity, Btu-ft ft ² -sec-°F	Melting point, °F	Camera average surface temperature near stagna- tion point, °F	Weight of model, grams				Condition of nose of model after tests	
								Initial	After first 50-second run	After second 50-second run	After third 50-second run		After fourth 50-second run
M-1	Silicon carbide, SiC, Crystolon "B"	Norton Co.	150	0.26	3.02×10^{-5}	4,712	2,340	13.439	13.370	13.366	13.359	13.352	Slight erosion.
M-2	Silicon carbide + carbon, SiC + C, (unglazed)	Alfred Univ.						10.461	10.188	10.155	10.141	10.104	Erosion, slight glaze.
M-3	Silicon carbide + carbon, SiC + C, (unglazed)	National Cruc.	112.4	0.18	2.315×10^{-5}		2,450	8.054	7.896	7.822	7.751	Model broken	Erosion, slight glaze.
M-4	Silicon carbide + carbon, SiC + C, (unglazed)	National Cruc.	112.4	0.18	2.315×10^{-5}		2,450	9.366	9.307	9.278	9.250	9.198	Erosion, slight glaze.
M-5	Silicon carbide + silicon, SiC + Si, "Dury"	Carborundum Co.	149.8	0.20	2.345×10^{-5}		2,250	14.538	14.528	14.528	14.539	14.528	Very slight erosion.
M-6	Silicon carbide-silicon nitride, SiC/Si ₃ N ₄	Carborundum Co.	168.6	0.30	9.525×10^{-5}		2,400	13.612	13.594	13.571	13.575	13.576	Slight erosion, slight glaze.
M-7	Silicon nitride, Si ₃ N ₄	Carborundum Co.	214.3	0.27	40.251×10^{-5}	3,452	2,400	12.323	12.307	12.307	12.311	12.317	Slight erosion.
M-8	Titanium carbide-nickel, 90TiC/10Ni, K-150A	Kennametal Inc.	350		3.89×10^{-5}		2,400	26.799	26.762	26.757	26.778	26.776	Light brown coat.
M-9	Titanium carbide-nickel, 80TiC/20Ni, K-151A	Kennametal Inc.	362	0.14	4.84×10^{-5}		2,400	27.008	27.015	27.012	27.014	27.001	Erosion, brown coat.
M-10	Titanium carbide-nickel, 60TiC/40Ni, K-151B	Kennametal Inc.	394	0.11	6.12×10^{-5}		2,350	29.417	29.402	29.390	29.187	29.048	Erosion and melting, tan scale.
M-11	Titanium carbide-molybdenum- nickel, 50TiC/50Mo-Ni, K-151B1	Kennametal Inc.	394	0.12	6.39×10^{-5}		2,360	30.938	30.927	30.848	30.743	30.656	Erosion and melting, brown scale.
M-12	Titanium carbide-molybdenum- nickel, 50TiC/50Mo-Ni, K-151B	Kennametal Inc.	412		6.39×10^{-5}		2,380	27.288	27.179	27.112	26.987	26.776	Much erosion and melting, brown scale.
M-13	Titanium boride, TiB ₂	Norton Co.	281		4.16×10^{-5}	5,252	2,360	19.855	19.871	19.894	19.912	19.911	Slight erosion, slight glaze.
M-14	Titanium boride, TiB ₂	Norton Co.	281		4.16×10^{-5}	5,252	2,360	18.770	18.790	18.801	18.799	18.809	Slight erosion, slight glaze.
M-15	Titanium boride, TiB ₂	Borolite Corp.	268				2,490	22.509	22.437	22.442	22.444	22.445	Slight glaze, yellow coat.
M-16	Titanium boride-boron carbide, 99TiB ₂ /1B ₄ C	Norton Co.	270		4.10×10^{-5}		2,260	20.633	20.636	20.638	20.639	20.639	Little change noted.
M-17	Titanium boride-nickel, TiB ₂ /Ni	Carborundum Co.					2,260	20.207	20.212	20.218	20.220	20.221	Adherent greenish coat.
M-18	Chromium boride, CrB ₂	Borolite Corp.				3,340	2,350	22.182	22.235	22.264	22.303	22.318	Slight glaze.
M-19	Chromium-alumina, 77Cr/23Al ₂ O ₃ , IT-1	Haynes Stellite Co.	374.6	0.16	8.06×10^{-5}	3,362	2,270	27.150	27.137	27.149	27.161	27.165	Adherent yellow coat.
M-20	Chromium-alumina, 72Cr/28Al ₂ O ₃ , Al ₆ 145	Ohio State University	375		8×10^{-5}		2,270	26.150	26.122	26.121	26.124	26.123	Adherent yellow-green coat.
M-21	Chromium-molybdenum-alumina, 50Cr-15Mo-35Al ₂ O ₃ , IT-1B	Haynes Stellite Co.	380.2	0.16			2,270	26.746	26.748	26.751	26.751	26.753	Adherent yellow-green coat.
M-22	Chromium-tungsten-alumina, 25Cr-40W/15Al ₂ O ₃ , IT-2	Haynes Stellite Co.	370.2				2,300	28.187	28.147	28.101	28.065	28.048	Erosion, dark green coat.

368 - 1,850° F
 372 - 2,150° F
 381 - 1,810° F
 400 - 2,400° F
 Not 500° F

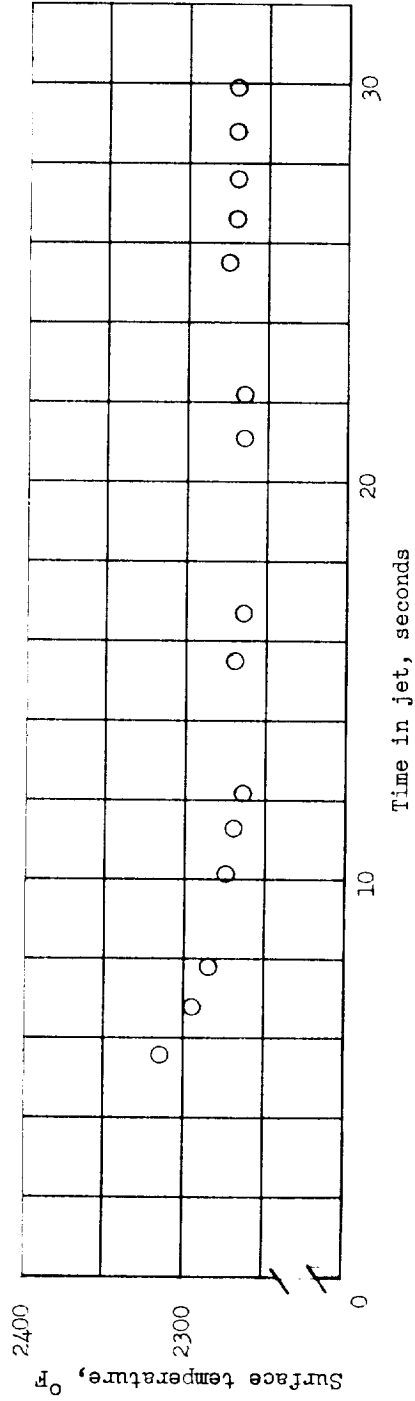


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Figure 1. - Typical 1/2-inch-diameter model during test in a Mach number 2 ceramic-heated air jet at a stagnation temperature of 3,800° F.



(a) Model M-7.



(b) Model M-19.

Figure 2. - Typical 2 run average camera surface temperatures near the stagnation point of 1/2-inch-diameter models plotted against time in the Mach number 2 jet at a temperature of 3,800° F.

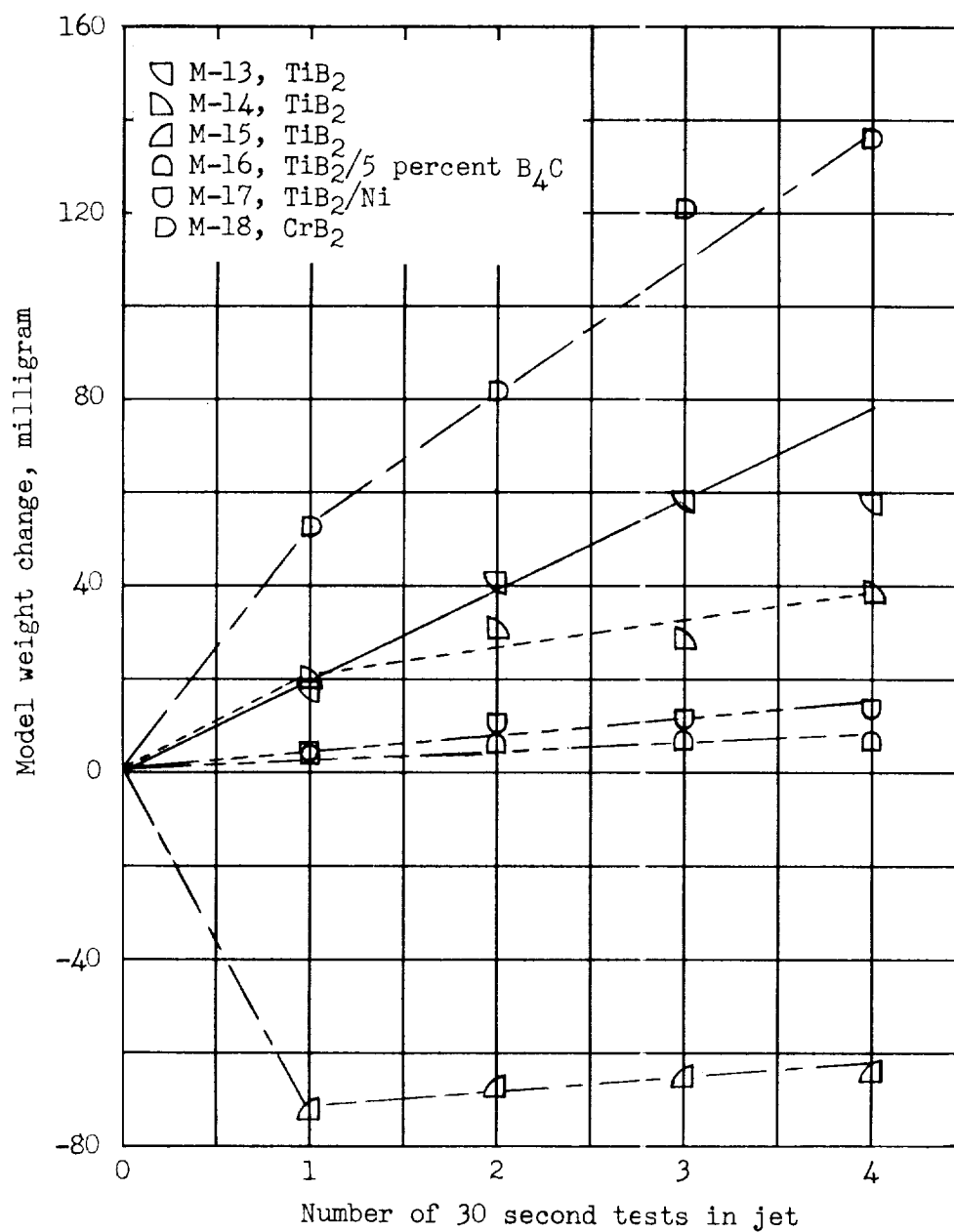


Figure 5.- Weight-change data for models of materials containing or consisting of metal borides.

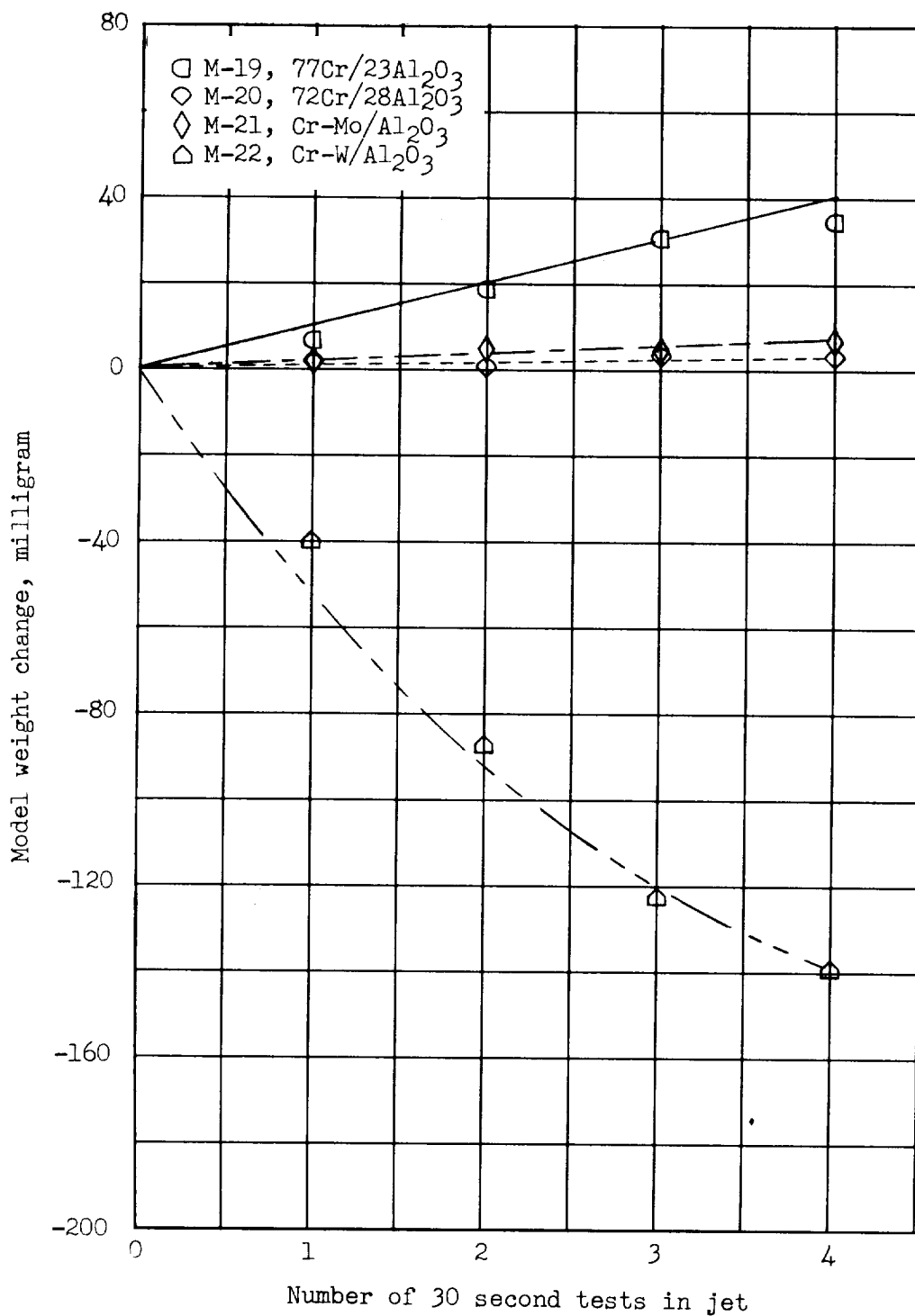


Figure 6.- Weight-change data for models of cermets containing Al₂O₃.

